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CERAMIC COATED BEARING ELEMENTS FOR IMPROVED DURABILITY AND RELIABILITY

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13. ABSTRACT *(Maximum 200 words)*

BIRL, of Northwestern University, conducted research in the application of hard, wear-resistant coatings onto bearing steels and silicon nitride. These coatings were deposited using the high-rate reactive sputtering (HRRS) process. Prior to this work, the authors had demonstrated the ability to deposit hard coatings such as titanium nitride onto materials such as M2 tool steel, cemented carbide inserts, and hardened 440C steel without degradation of the metallurgical properties of the coated piece. In this program, it was shown that TiN coatings deposited onto test components, which simulate entire hybrid bearings, had extended the average rolling contact fatigue (RCF) lifetimes of the uncoated components by factors of at least 2.6 on average and as much as 4.6 for specific components when run under accelerated conditions. Since the M50 material failed in many of these tests, it is believed that non-accelerated tests would show even larger increases in RCF lifetime due to the coatings. Accelerated test results suggest the increases in RCF lifetime should be obtainable on real-life components coated with thin, hard materials.

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FOREWORD

This document is the final report covering the work performed under U. S. Air Force Systems Command Contract F33615-92-C-5935. The project was sponsored by the Materials Directorate, Wright Laboratory, Air Force Systems Command, Wright Patterson AFB OH 45433-7750. The Advanced Research Projects Agency (ARPA), Arlington VA, was the original source of the funding. The Air Force Project Engineer was Karl R. Mecklenburg of Wright Laboratory.

1. EXECUTIVE SUMMARY

BIRL, the industrial research laboratory of Northwestern University, has conducted unique and innovative research, under sponsorship [1] from the Advanced Research Project Agency (ARPA), in the application of hard, wear-resistant coatings onto bearing steels and ß-silicon nitride (ß-Si_3N_4). These coatings were deposited using the high-rate reactive sputtering (HRRS) process that was pioneered by Dr. William Sproul [2], the principal investigator of this program. Prior to this work, Dr. Sproul and co-workers had demonstrated [2-5] the ability to deposit hard coatings such as titanium nitride (TiN) onto materials such as M2 tool steel, cemented carbide inserts, and hardened 440C steel without degradation of the metallurgical properties of the coated piece. In this program, it was shown that TiN coatings deposited onto test components, which simulate entire hybrid bearings, had extended the average rolling contact fatigue (RCF) lifetimes of the uncoated components by factors of at least 2.6 on average and as much as 4.6 for specific components when run under accelerated conditions. Since the M50 material failed in many of these tests, it is believed that non-accelerated tests would show even larger increases in RCF lifetime due to the coatings. Even the accelerated results suggest the increases in RCF lifetime that should be obtainable on real-life components coated with thin, hard materials.

Specifically, this ARPA program at BIRL had the objectives to

- Use hardened M50 and ß-Si_3N_4 flat coupons for the optimization of TiN, titanium aluminum nitride ($Ti_{0.5}Al_{0.5}N$), and chromium nitride (CrN_x) deposition parameters for best coating properties and adhesion;
- Coat 0.5-inch-diameter hardened M50 and ß-Si_3N_4 balls for rolling contact fatigue (RCF) testing with the previously mentioned materials;
- Coat 0.375-inch-diameter hardened M50 and ß-Si_3N_4 rods for Federal-Mogul Weibull tests;
- Measure the uniformity of the thickness of these coatings on the balls and rods, which will be in the range of 0.2 to 1.0 μm;
- Coordinate with other ARPA program contractors for the evaluation of the coatings on test specimens and bearing elements.

Overall, the objectives of the ARPA program have been successfully met. All of the coatings have been produced using the HRRS process to control the coating composition, balls for RCF have been coated, and a technique has been developed for measuring coating thicknesses on curved surfaces. Details of the coating processes, characterization results of the various materials, thickness measuring technique, and RCF testing are presented in the main body of this report.

The three coatings were deposited onto M50 and ß-Si_3N_4 0.5-inch-diameter balls in thicknesses of 0.3 to 1.0 μm. The RCF testing proved an effective screening tool for the development of these coatings. Under extremely severe operating conditions (5400 revolutions·min^{-1} (rpm) and 6.8 GPa (990 ksi) Hertz contact pressure), the average RCF lifetime increased by a factor of at least 1.3, while specific tests showed almost 5 fold increases. The greatest improvement in RCF life was achieved with 0.75-μm- thick TiN coatings where the lifetimes increased by a factor of 2.6 on average, and 4.6 for two specific examples. CrN coatings of the same thickness yielded a 2 fold increase in lifetime, while 0.5- and 1.0-μm-thick $Ti_{0.5}Al_{0.5}N$ coatings showed increases of 1.3. We were able to successfully coat ß-Si_3N_4 material with good coating adhesion and enhanced RCF lifetimes, which suggests the viability of a hybrid bearing in which both the metal and ceramic are coated.

2. COATING DEPOSITION

2.1 Experimental Procedures

All of the coating work was performed in two sputtering systems, shown schematically in Figures 1 and 2. Most of the developmental work was performed in the single-cathode, balanced magnetron 902M Materials Research Corporation (MRC) in-line sputtering system (Figure 1). Similarly, most of the coatings of balls for RCF testing, and all of the rods for Federal-Mogul Weibull testing, were coated in BIRL's opposed-cathode, unbalanced magnetron sputtering system (Figure 2). High purity argon (99.999%) was used as the sputtering gas, and high purity nitrogen (99.9995%) was the reactive gas for all coatings. Automatic feedback control was used to maintain the nitrogen partial pressure at the desired set point during the depositions. The control system consisted of MKS mass-flow controllers coupled with a quadruple mass spectrometer (either an UTi for the single cathode unit or an Inficon Quadrex 100 on the opposed cathode unit) to achieve automatic feedback control of the reactive gas [6]. MRC Mu Inset™ shaped sputtering targets were used in this program, and the materials were all 99.94% or better purity.

The titanium-aluminum targets were bought for a previous program, and were intended to be a 50-50 atomic percent material. Analysis of the target after the work was performed in this program identified the material as 50-50 weight percent (about 64 atomic percent aluminum). This does not alter the results of this work, but perhaps offers a reason for discrepancies between our characterization results and those of other researchers. It must be added that, although the 50-50 weight percent material is a two-phase composition, our X-ray diffraction analysis of the coatings indicated only single-phase material in the nitride form. Also, semi-quantitative analysis performed with the scanning electron microscope showed the coatings to be roughly 50-50 atomic percent. Throughout this report, the notation "$Ti_{0.5}Al_{0.5}N$" will refer to weight percent, unless otherwise stated.

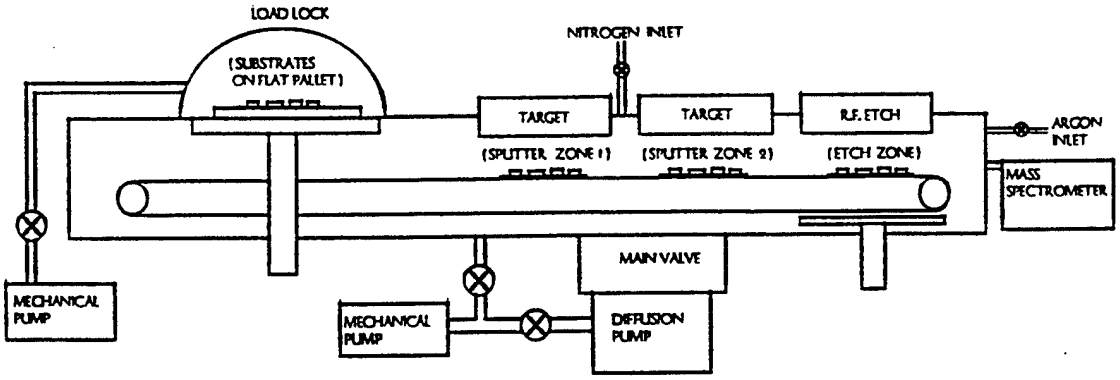

Figure 1. Schematic drawing of the single cathode magnetron sputtering system

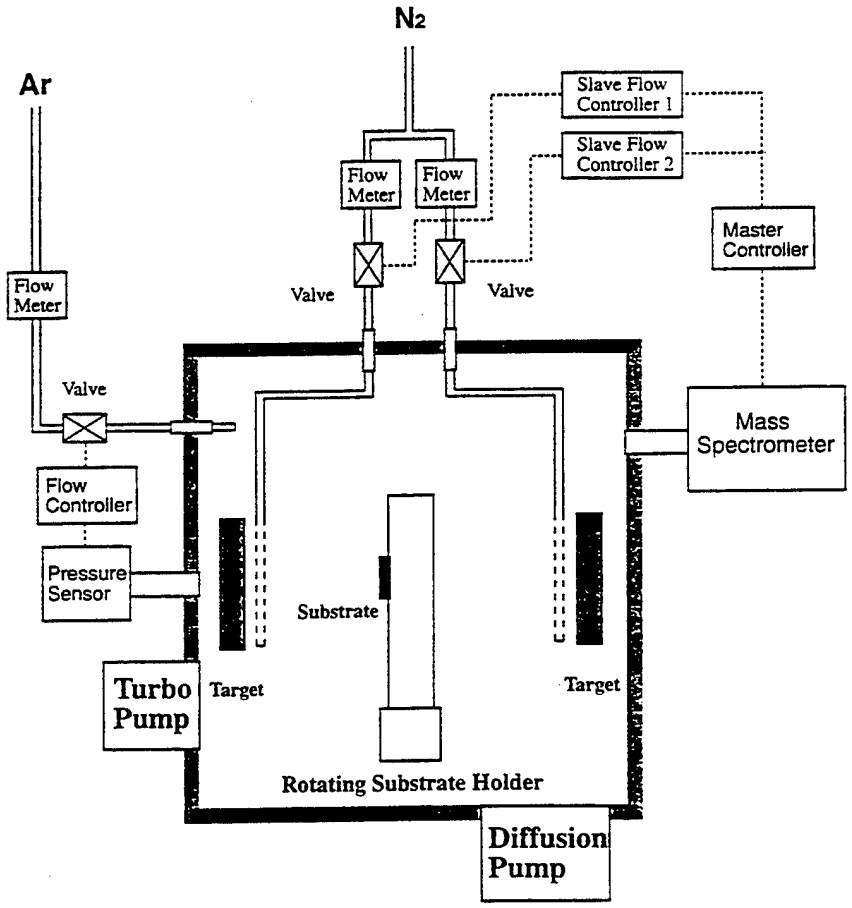

Figure 2. Schematic drawing of the opposed cathode unbalanced magnetron sputtering system

Two different types of substrates were used in this program. Rectangular test coupons (1.2 x 2.5 x 0.6 cm) of M2 tool steel (Doall Corporation) and square ß-Si_3N_4 inserts (1.2 x 1.2 x 0.6 cm) (Norton Diamond Film Company) were used as substrates in the development of the TiN and $Ti_{0.5}Al_{0.5}N$ coatings. M2 steel was used because of our experience with it and because of difficulty obtaining properly hardened M50 NiL material within schedule. Once the parameters for the desired coating had been established, balls of hardened M50 (NTN) and ß-Si_3N_4 (Cerbec NBD-200) were coated for the RCF testing. The balls were all 0.5 inch in diameter. The average roughness (Ra) of the M50 balls was 2 to 4 μinches (500 to 1000 Å), while that of the ß-Si_3N_4 was less than 0.15 μinch (37 Å). The rods were 3 inches long, and 0.375 inch in diameter. Prior to being placed in the sputtering chamber, all of the substrates were ultrasonically cleaned in acetone and then alcohol.

In the opposed-cathode system, rectangular test coupons were mounted at the midpoint of a vertically placed cylindrical substrate holder (7.5 cm dia. x 38cm high), which typically rotated at a speed of 10 rpm during deposition.

The RCF balls were held in basket holders that were mounted in a fixture with two-fold ("planetary") rotation. The height of the balls was the same as the test pieces, roughly the midpoint of the sputtering targets. The planetary fixture was run with a 10 rpm rotation speed, and for each turn of the fixture, the sample holder turned 1.33 times. In the single-cathode system, the flat coupons were placed in the center of a 31 x 31 cm square pallet, and a self-contained rotary fixture held the rods and/or a basket in which the balls were placed. The rotation speed of this device is typically about 2 rpm.

Once the opposed-cathode sputtering system was pumped down to a base pressure of 2.7 x 10^{-4} Pa (2 x 10^{-6} Torr) or less, it was backfilled with argon to a working pressure of 0.27 Pa (2 mTorr), and the samples were given an rf etch for 10 minutes with an rf etch power of 1.0 kW. After the etch, the argon pressure was changed to 1.07 Pa (8 mTorr), power was applied to the targets and substrate, the reactive gas (nitrogen) was introduced, and the deposition was begun. The target power, substrate bias voltage, nitrogen partial pressure, and total pressure were maintained constant throughout the run.

The coating sequence was similar in the single-cathode system. When the chamber reached a base pressure of 6.6×10^{-5} Pa (5×10^{-7} Torr) or less, the chamber was backfilled with argon to a pressure of 1.07 Pa (8 mTorr) for test coupons or 0.27 Pa (2 mTorr) with the rotation device. The rotation samples were given a 5-minute rf etch at 1.5 kW. The coatings had better adhesion to the ß-Si_3N_4 substrates when the etch time was 20 minutes, but the rotation device is not capable of operating for that long. Once the etch was finished, the samples were moved from the etch station to a position under the target. No scanning motion was used while the samples were being coated, but the baskets were rotated during the coating.

Coating thickness was controlled by the length of time during the deposition, and deposition rates were established by measuring the thickness with the Calotest on flat specimens coated for a specific length of time. For most hardness and adhesion tests, coatings between 3 and 5 μm thick were deposited onto the rectangular coupons.

The coatings were characterized for hardness, adhesion, and crystal structure. The hardness of the coatings was measured using a Leco DM-400FT microhardness tester equipped with flat-field optics and a magnification up to 1000x. The adhesion of the coatings was measured with a manual CSEM Revetest, using indentors with a radius of 200μm. The crystal structure was of the coatings was determined using a Scintag PAD V 2000 X-ray diffractometer with Cu Kα radiation.

2.2 Coating Results

All three coating materials, TiN, $Ti_{0.5}Al_{0.5}N$, and CrN_x, had been reactively sputtered in these units in previous work on M2 and 440C steels [5], but there existed only limited experience with coating ceramics, such as ß-Si_3N_4. A wide variety of operating parameters were examined throughout the course of this program. The operating conditions used for the final rolling-four-ball tests are listed in Table 1 and represent the deposition conditions that produced the best combination of hardness and adhesion. The hardness and scratch test critical load values are from thicker coatings deposited onto test blocks.

Table 1: The Deposition Conditions, Hardness, Scratch Test Critical Load, and Lattice Parameter of the Coatings

Operating Parameters			Coating Material		
			TiN	$Ti_{0.5}Al_{0.5}N$	CrN
Etch Conditions	Unit		Single	Opposed	Opposed
	RF Etch Power, kW		1.3	1.0	1.0
	Total Pressure, Pa (mTorr)		1.07 (8.0)	1.07 (8.0)	1.07 (8.0)
	Etch Time, minutes		5	10	10
Deposition Conditions	Target Power, kW		10	5 each	2.5 each
	Pressure, Pa (mTorr)	Total	1.07 (8.0)	1.07 (8.0)	1.07 (8.0)
		Nitrogen Partial	0.024 (0.18)	0.033 (0.25)	0.133 (1.0)
	Negative Substrate Bias, V		150	95	125
Coating	Hardness, $kgf \cdot mm^{-2}$		2500	1800	2000
	Critical Load, kgf/ Thickness, μm		5.0/4.5	5.0/2.4	6.0/8.0
	Lattice Parameter, Å		4.25	4.21	4.19

As it turned out, there were differences in three of the operating parameters for the different coatings: target power, substrate bias voltage, and nitrogen partial pressure. For the TiN and $Ti_{0.5}Al_{0.5}N$ coatings, the total target power is the same, 10 kW, and the nitrogen partial pressures are very similar, 0.024 and 0.033 Pa (0.18 and 0.25 mTorr), respectively. The considerable difference between the substrate bias voltages used is partially sputtering system dependent: initial TiN coatings in the opposed cathode system used a substrate bias of -90V. Due to the different magnetron configurations in the two units, lower substrate bias voltages are needed in the opposed cathode unit to obtain fully dense films.

The biggest changes in target power and nitrogen partial pressure were for the CrN. Some of the difference was due to taking advantage of CrN_x studies that had been performed on 440C substrates. These studies had shown very good hardness and adhesion results for coatings greater than 5 μm in thickness. However, the power had been reduced to 2.5 kW to minimize the risk of overheating and, consequently, softening the substrate. The higher nitrogen partial pressure needed to produce stoichiometric CrN is due to the lower affinity of Cr to nitrogen than for Ti. In these studies, substrate bias voltages lower than -125 V yielded coatings with hardnesses on the order of 1400 $kgf \cdot mm^{-2}$ or less.

The lattice parameters for all three coatings are also shown in Table 1 and were calculated from the patterns shown in Figure 3. All of the parameters are larger than the bulk values reported in the JCPDS files [7] but are comparable to literature values [8 -10]. These x-ray scans were performed using Bragg-Brentano geometry, so the diffraction planes are parallel to the surface of the sample. If the surface is in a state of compression, the distance between planes increases. Thus, a compressive strain gives a larger lattice parameter. The expansion in the lattice parameter is probably due to high compressive residual stress in the films. At BIRL, X-ray residual stress measurements have only been made on TiN coatings, but these verified the high levels of stress, typically on the order of -4 to -10 GPa.

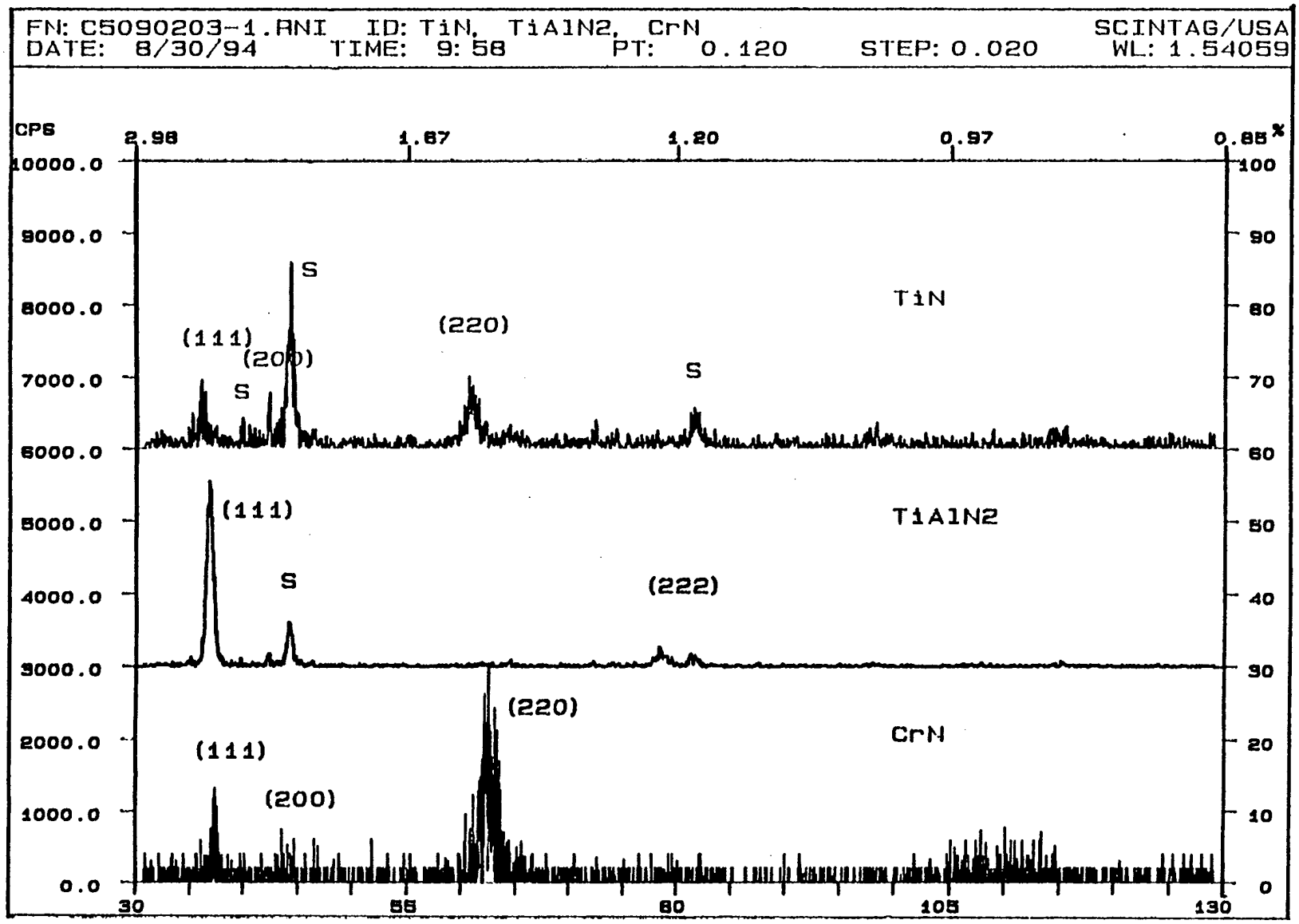

Figure 3. X-ray diffraction patterns of the coatings. X-axis is the diffraction angle (°2θ). Y-axis is the intensity in arbitrary units.

As seen from the patterns, the orientation of the coatings varied considerably, even though the materials are all B1 (NaCl) type. TiN has a combined (111) and (220) orientation and sometimes almost random, while $Ti_{0.5}Al_{0.5}N$ is highly (111) oriented, and most of the CrN coatings produced were (220) oriented.

3. MEASUREMENT OF COATING THICKNESS

Measurement of the coating thickness is an important aspect of coating characterization because of its direct influence upon the values obtained for other properties, such as hardness and scratch adhesion critical loads. Many methods exist for measuring coating thickness, such as cross-sectioning, Calotesting, and even weight differential methods, but none can provide the sensitivity and reliability needed for measuring approximately 1.0-μm-thick coatings on a curved surface. None of them addresses the issue of uniformity of the coating, which will be particularly important in a rolling element because the entire surface is a candidate for use. Energy dispersive spectroscopy has proved useful in addressing both of these issues.

3.1 Energy Dispersive Spectroscopy

Energy dispersive spectroscopy (EDS) in the scanning electron microscope (SEM) was used to determine the uniformity of coatings by measuring the intensity of the iron signal from coated steel balls. Six points on the coated sample were designated for examination: four equidistant points along the "equator" and the "North" and "South" Poles. Use of this technique has provided us with the information to reduce the typical intensity variation of the iron signal from ±15% to ±6% or less. This small intensity variation was found to be reproducible from run to run, regardless of sample position.

To create calibration standards, polished M2 steel flats were coated in the single cathode system with TiN in nominal thicknesses of 0.5, 0.75, and 1.0 μm. The deposition conditions were similar to those listed in Table 1, except that the substrate bias voltage was -100V.

The thicknesses of the TiN coatings were measured using the Calotest, a ball-cratering device typically used for measuring thicknesses on flat specimens. These samples, as well as an uncoated M2 piece, were then examined using EDS, and the intensity of the titanium and iron signals were recorded. When the Ti EDS intensities are plotted versus the measured Calotest values, the coating intensities increase linearly with thickness because more material is being sampled as the thickness increases. The EDS iron intensities should decrease

exponentially with increasing thickness because the iron signal from the substrate is dependent upon the amount of the absorbing material present, and absorption of x-rays is an exponential function. Therefore,

$$I_{Ti} = a + bx \qquad \text{and} \qquad I_{Fe} = c \cdot \exp(dx)$$

where I_{Ti} and I_{Fe} are the intensity of the titanium and iron signals, respectively, x is the thickness of the coating in microns, and a, b, c, and d are constants.

The plots of I_{Ti} and I_{Fe} versus the Calotest thicknesses are shown in Figure 4. The fit of the I_{Ti} data is linear, whereas the one for I_{Fe} is exponential. Both curve fits have an R^2 factor, which is a measure of confidence, of more than 0.992, where 1.000 is considered an exact fit.

Balls made of 52100 steel were coated in the basket using the parameters described above to produce nominally 0.75-μm-thick coatings. Examination of the EDS spectra showed iron intensities of 1800 counts, with titanium intensities of about 3000 counts. These data were inconsistent with the calibration information presented in Figure 4. For example, if the values for I_{Fe} were correct, the coating was 0.25 μm, whereas if the I_{Ti} values were correct, the coating was 0.7 μm thick. The iron signal of an uncoated 52100 ball was virtually identical to that of the M2 steel, so the change in steel did not cause this discrepancy.

To understand this phenomenon, an M2 steel flat and a 52100 ball were placed in the basket and coated without rotation, which permitted the thickness on the M2 to be directly compared, using both the EDS and Calotest methods. Three different coating thicknesses were deposited. Analysis of the thickness vs. iron signal results show that the iron intensities from the pieces coated in the basket fit a different curve than the original M2 calibration curve. The values of I_{Fe} fit a second order curve, such that $I_{Fe} = a + bx + cx^2$, where x is the thickness and a, b, and c are constants. The R^2 factor for this second order fit was 0.999. The two iron calibration curves are shown in Figure 5.

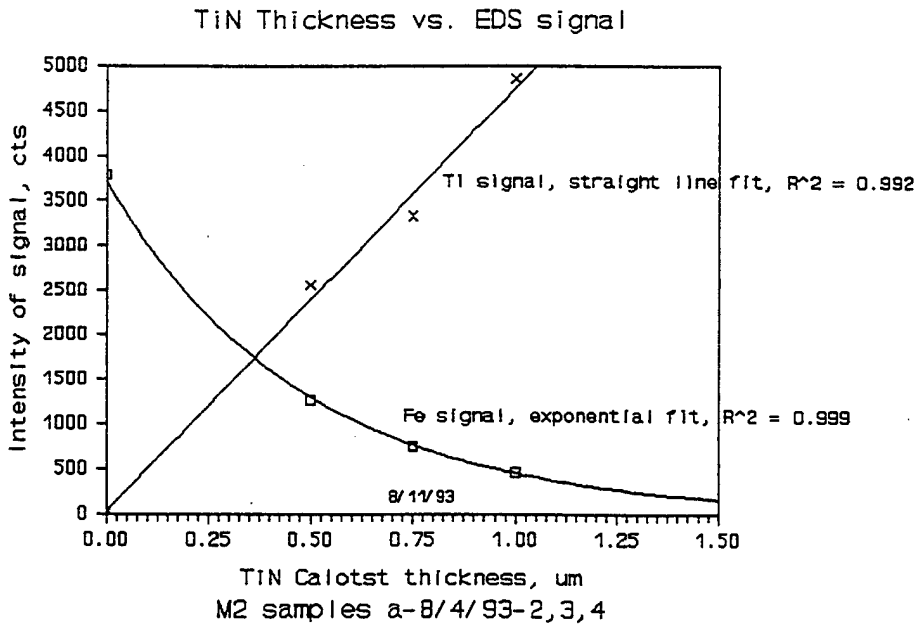

Figure 4. Titanium and iron EDS signals for TiN coated M2 steel calibration pieces.

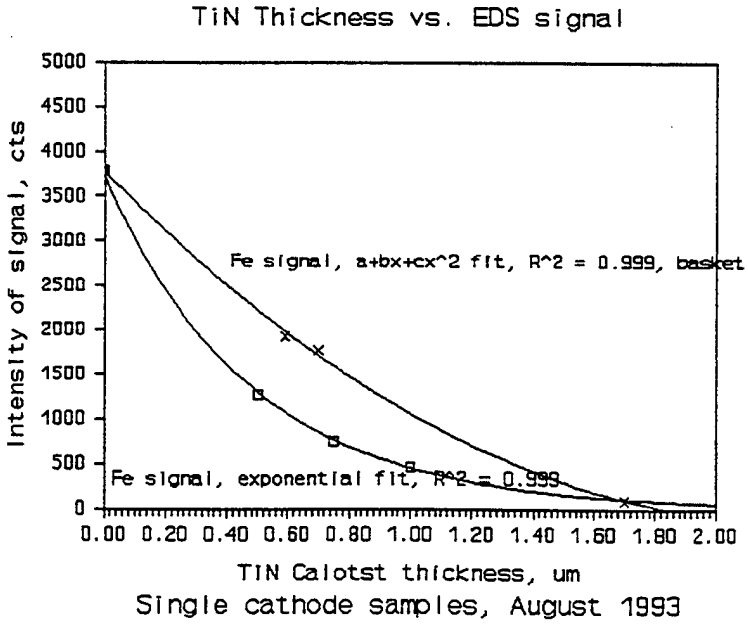

Figure 5. EDS iron signals from M2 pieces coated in the basket (second order fit) and the calibration pieces

Although there is no physical reason why the iron signals should fit a second order curve, it is not unique. Similar TiN thickness vs. iron signal experiments from samples made in BIRL's four-cathode ABS™ sputtering unit demonstrate the same phenomenon, with an R^2 of 1.000. The precise reason for the second order fit is unclear, although it is at least partially a function of the density of the coating. (The coating density should also affect the values for I_{Ti}, but this primarily changes the slope of the line fit to the data.) This can be concluded because the ABS™ samples were run at lower-than-normal bias voltages, which causes the coating to be less dense than normal. Also the basket in the single cathode unit interferes at least intermittently with the line-of-sight nature of PVD coating, which may cause a decrease in coating density. Balls coated inside the basket are darker than those made outside the basket under the same conditions. This seems to indicate that the basket affects the coating process in other ways.

An M2 steel piece was then coated in the basket at -150 Volts substrate bias. The measured value of I_{Fe} for this sample was closer to the calibration curve than the -100 Volt samples, although it was still larger than the calibration curve results. M50 balls for rolling-four-ball tests were coated using substrate bias voltages of -100 and -150 Volts, with all other operating parameters remaining the same. The specimens coated at -150 Volts substrate bias were expected to be more dense than those coated at -100V, and therefore perform better. The average RCF lifetimes of these samples were determined using test conditions of 4800 rpm and 5.84 GPa (850 ksi) with a coated M50 drive ball against uncoated M50 driven balls and showed an improvement from 7.2 hours with a standard deviation, σ_{n-1}, equal 0.1 hours to an average of 11.2 hours with σ_{n-1} equal 3.3 hours for the drive balls coated at -100V and -150V, respectively. Thus, although the process was not optimized, the thickness technique provided information that led to improvements in the coating quality and performance.

3.2 $Ti_{0.5}Al_{0.5}N$ Thickness Calibration

Similar experiments were performed for $Ti_{0.5}Al_{0.5}N$ coatings in the opposed cathode system. For these studies, two M2 steel pieces were coated in each deposition cycle: one inside a basket ("caged") and one without a basket ("uncaged"). This not only permitted thickness calibration to be performed, but also allowed us to compare the hardness and adhesion of the coatings made inside and outside of a basket. The two-fold rotation fixture was used during this study, in order to emulate the results that we would observe on the balls for RCF tests.

The operating parameters and characteristics for these $Ti_{0.5}Al_{0.5}N$ coatings were listed in Table 1. Using two-fold rotation, these characteristics were altered significantly. The sample coated without a basket showed a Vickers hardness, H_V, of 2800 $kgf \cdot mm^{-2}$ and a critical load, L_C, of 2.0 kgf for a coating thickness of 3.1 μm. Although harder than the coatings made on samples with a single rotation, the critical load of this coating was also lower. By comparison, the coating on the M2 sample in the basket had a thickness of 2.4 μm, a H_V of 1800 $kgf \cdot mm^{-2}$, and an L_C value of 5.0 kgf. This was the first $Ti_{0.5}Al_{0.5}N$ coating that demonstrated a critical load value greater than 3.0 kgf.

Intensity plots of I_{Ti} and I_{Fe} versus the Calotest thicknesses are shown in Figure 6, for both the caged and uncaged calibration specimens. The fits of the I_{Ti} data are linear, whereas those for I_{Fe} are exponential. All of the fits to the data sets have an R^2 factor of more than 0.950.

The calibration procedure indirectly revealed the reason for the differences in hardness and adhesion results between caged and uncaged specimens mentioned above. Figures 7 and 8 show the EDS spectra for the uncaged and caged samples from 9/29/93-2. The uncaged sample demonstrates the same Ti:Al ratio, about 1.3:1, as the target, which is 50 weight percent aluminum. The coating made inside the basket has a Ti:Al ratio of about 1:1, however. Based upon analysis of these spectra, the coatings inside the basket are about 54 weight percent aluminum. Samples made with the single rotation fixture also showed a

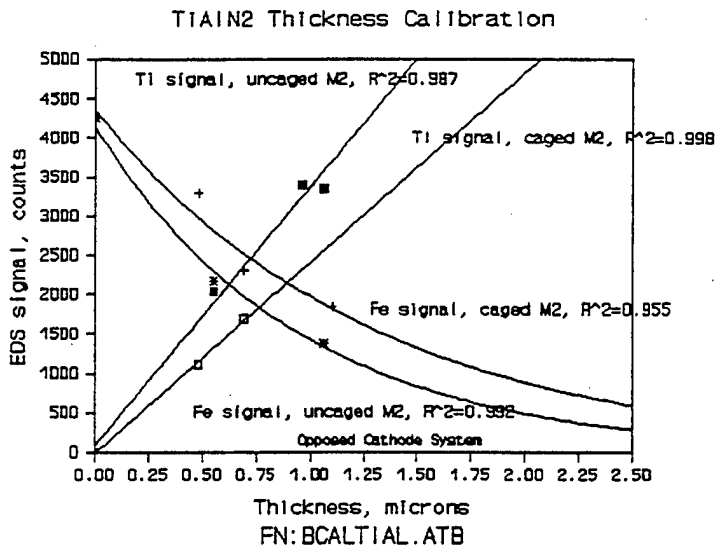

Figure 6. Titanium and iron calibration curves for caged and uncaged $Ti_{0.5}Al_{0.5}N$ coated M2 samples.

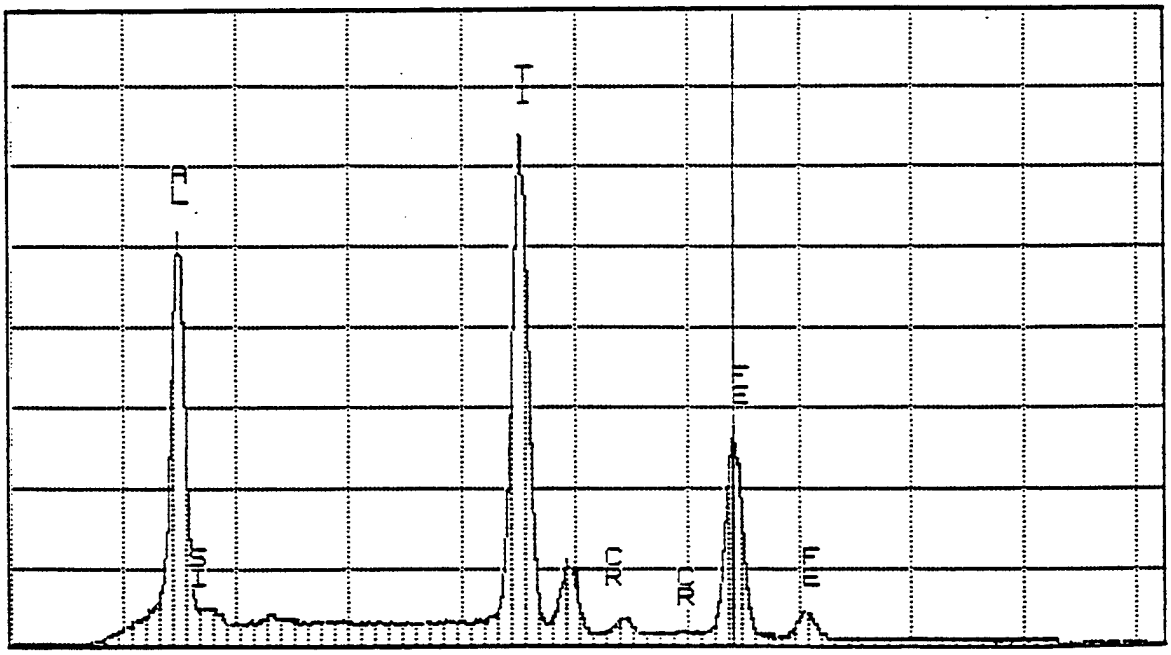

Figure 7. Energy dispersive spectroscopy (EDS) spectrum for uncaged $Ti_{0.5}Al_{0.5}N$ coated M2 steel sample. X-axis range is 0 to 10.24 keV. Y-axis range is 0 to 4096 counts.

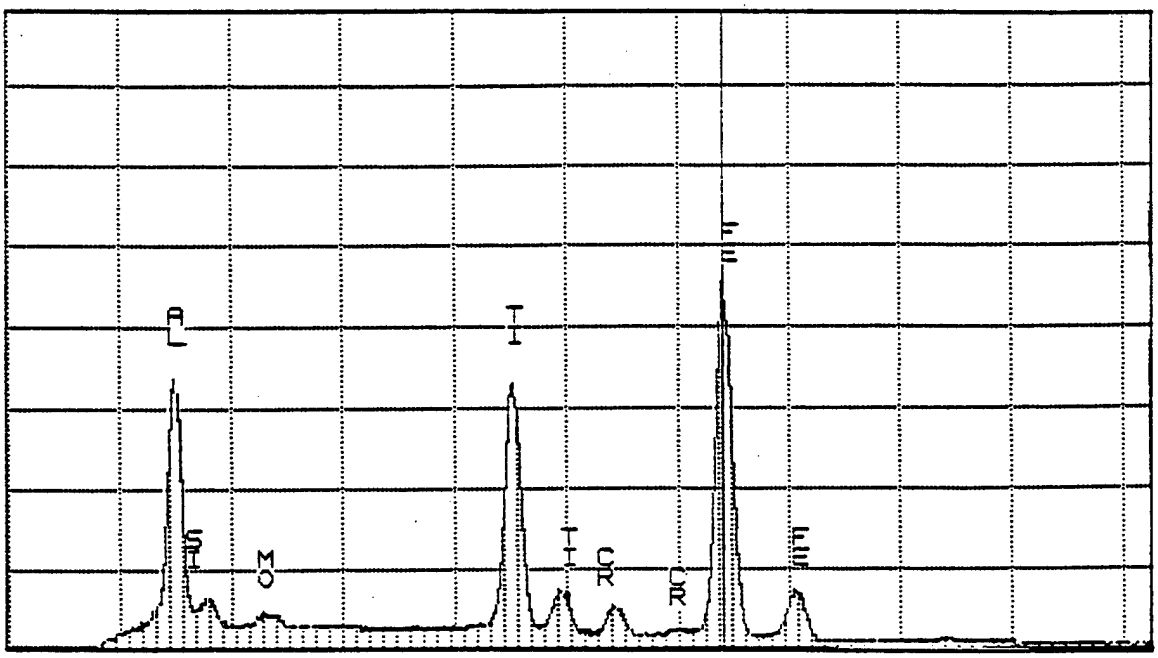

Figure 8. Energy dispersive spectroscopy (EDS) spectrum for caged $Ti_{0.5}Al_{0.5}N$ coated M2 steel sample. X-axis range is 0 to 10.24 keV. Y-axis range is 0 to 4096 counts.

Ti:Al ratio of 1.3:1. The ratio of the intensity of the aluminum signals between the uncaged and caged samples is the same as the ratio of the thicknesses of these coatings, about 1.54:1. This also explains the difference in the Ti signals shown in Figure 6, which did not agree, even after compensating for thickness.

The XRD patterns of these samples also differ, as shown in Figure 9. The unlabelled peak in both cases is the $Ti_{0.5}Al_{0.5}N$ (111) diffraction peak. The pattern for the uncaged sample is very similar to those for coatings made using only single rotation, with a lattice parameter of 4.21 Å and a full-width at half-maximum (FWHM) of about 0.55 degree 2θ. The caged sample has a lattice parameter of 4.18 Å and a FWHM value of 0.15 degree 2θ. The smaller lattice parameter, as seen in the caged coatings, normally indicates smaller compressive stresses in the coating. These smaller compressive stresses contribute to the lower hardness values of the caged coatings relative to the uncaged ones. However, the smaller lattice parameter may be due to the

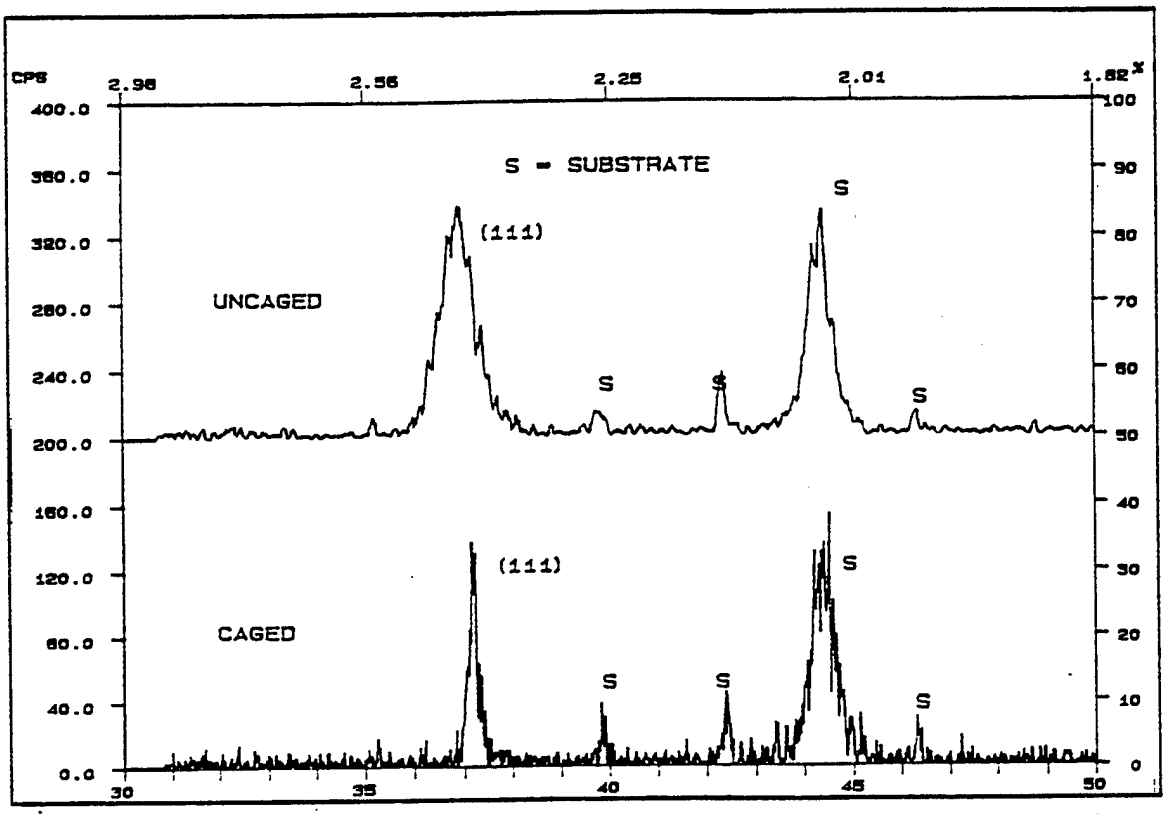

Figure 9. X-Ray Diffraction patterns for samples 9/21/93-1 uncaged and caged. The x-axis is degrees 2θ, and the y-axis is in intensity (arbitrary units).

smaller aluminum atoms replacing the titanium atoms, as has been previously seen in aluminum-rich "$Ti_{0.5}Al_{0.5}N$". The long-range order in such cases is still preserved. The increased broadening of the peaks from the uncaged coating, as illustrated by the larger FWHM value, could be attributed to either nonuniform strains or smaller grain sizes. Due to the low intensities of the higher order diffraction peaks of the coatings, it is difficult to determine the primary contributor to the broadening effect.

Both the smaller lattice parameter and reduced broadening in the caged coatings can be attributed to reduced ion bombardment within the basket. When the ion bombardment is lowered, the coating becomes less strained. Even though the current density varies at different points throughout the chamber, it is likely that the variation is not as great within the basket as outside of it. This would lead to more uniform strains and the smaller FWHM value. The aluminum atoms replacing the titanium atoms could also reduce the strain in

the unit cell. Although it is not understood why the titanium depletion occurs, coatings that are titanium depleted have shown better scratch adhesion results on M2 steel substrates than the $Ti_{0.5}Al_{0.5}N$ coatings. Why the titanium depletion occurs, as well as its effect upon the coatings, needs to be investigated further.

3.3 Uniformity

Throughout this project, uniformity of the coating on the substrates has been an important issue. Information from the EDS technique, with or without calibration standards, has driven modifications of the fixtures. It has prompted us to examine closely the path a substrate takes within the chamber as it is being coated in order to insure that all samples move through the same regions equally. Much developmental work has taken place due to these data, and more is required to fully understand and implement this information.

During the $Ti_{0.5}Al_{0.5}N$ calibration work, improvements were made to the ball-coating fixture that improved the uniformity of the coating on a ball from ±11% to about ±6% or less of the average value. The fixture now incorporates wobble, as well as the two-fold rotation. An example of the uniformity of the coating thickness on a $Ti_{0.5}Al_{0.5}N$ coated ball is shown in Figure 10.

Therefore, although originally considered a technique for measuring the uniformity of the coatings and, with calibration standards, the coating thickness, the use of the EDS has provided much more. It supplied information that led to increasing the density of the TiN coatings and subsequently improving the RCF lifetime. It also provided data that led to a better, although not complete, understanding of the improved $Ti_{0.5}Al_{0.5}N$ coating quality within a basket.

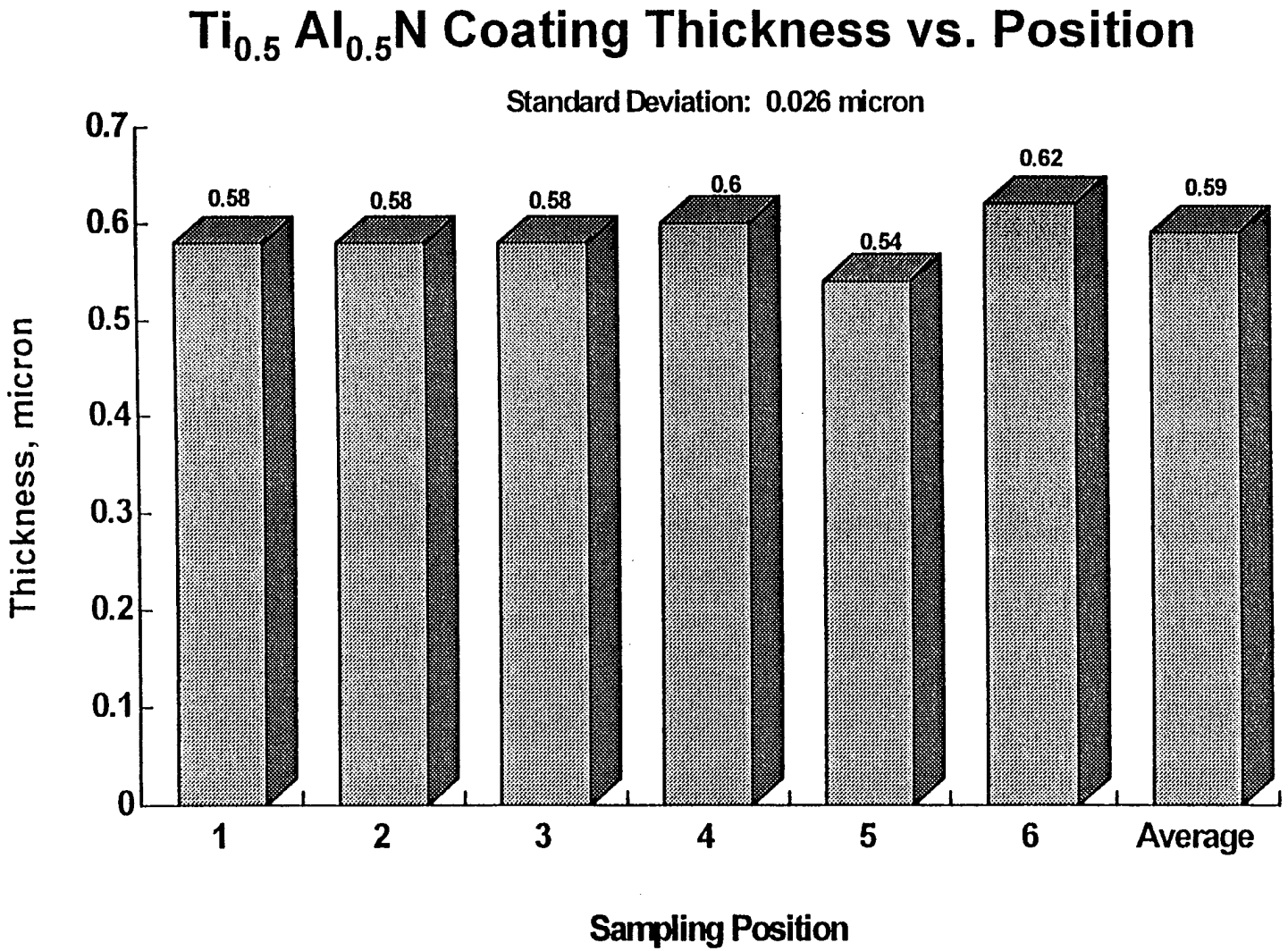

Figure 10. Uniformity of $Ti_{0.5}Al_{0.5}N$ coating thickness on a 52100 steel ball.

4. ROLLING CONTACT FATIGUE TESTING

All of the rolling-four-ball RCF testing was performed at BIRL under the direction of Dr. Peter Chang using BIRL's Falex tribology tester. The three-ball-on-rod RCF testing is due to be performed at Torrington under the direction of Dr. Phil Pearson. The RCF rods will be coated in the opposed-cathode sputtering system in the near future. After these rods are coated they will be sent to Torrington for testing.

4.1 RCF Test Procedures

A rolling four-ball adaptor for BIRL's Falex tribological test machine was used to provide RCF evaluation of the baseline materials and coatings. This adaptor consists of four balls of 0.5-inch diameter within an equilateral tetrahedral configuration. The upper rotating ball contacts and drives the three lower support balls to spin. The support balls are positioned 120° apart. The lower balls also rotate around a lower race cup, which restrains the balls from deviating from the adapter. Load is applied to the top ball as it is rotated at a given speed. The top ball is the drive ball, while the other three are the driven balls. For the hybrid bearing simulation, the drive ball is M50 steel (either coated or uncoated), while the driven balls are uncoated ß-Si_3N_4. In addition, an oil recirculating system was built to provide a continuous flow of Mobil D.T.E. lubricant to the test pieces, and the inlet lubricant temperature was about 24°C (ambient). The normal operating vibration level is about 0.05G, and the end of the rolling-four-ball test occurred when the measured vibration level went above 0.5G. When this pre-set value was exceeded, indicating the occurrence of a surface pit or fatigue spall, the Falex machine was automatically stopped. The test duration was also recorded by the Falex software. This enabled the calculation of the fatigue life cycles of the specimens being tested.

In the rolling-four ball test, failure is more common in the drive ball than in the driven balls because the former undergoes more contact stress cycles per revolution. We have made it a point <u>not</u> to replace failed driven balls, as is common practice, because we have been concerned with measuring failure

in the entire four-ball system. Although doing so does not give as accurate an indication of the performance of the coating (the drive ball is coated, except in the baseline tests), it is more representative of real life.

In order to produce RCF results, i.e., failure of the test components, within a reasonable time frame, values for two parameters were determined: test load and rotational speed of the drive-ball. The RCF tests can be accelerated by increasing both parameters, but caution must be taken. The test load generates a Hertzian contact pressure which must not cause any fracture or failure in the test balls. If this occurs, then the material yield strength has been exceeded. The rotational speed of the upper drive ball must not be so large as to lose the three lower orbital balls due to the effect of high centrifugal forces.

4.2 Hybrid Bearing RCF Results: ß-Si_3N_4 Drive Ball

The first set of hybrid RCF tests was performed using 0.3 μm thick TiN coatings made in the opposed cathode unit before the thickness measurement technique had been developed. By that time, the use of EDS to estimate uniformity had already caused improvements in the fixturing, such that the variation in EDS signal was reduced to ±15%. The balls were all coated in the same run, using two basket fixtures that had two cells each with one ball per cell.

For the initial RCF tests, TiN coated, as well as uncoated, ß-Si_3N_4 balls were used as drive balls against three uncoated M50 balls. These RCF tests were run using a rotational speed of 4800 revolutions per minute (rpm), and a Hertz contact pressure of 5.8 GPa (851 ksi), and the tests were operated under laboratory temperature (25°C) lubricated conditions. Although in practice it is unlikely that the ß-Si_3N_4 would be coated, these tests were performed in order to fulfill contract requirements.

The RCF lifetime results are shown in Table 2, and the ß-Si_3N_4 coated balls showed a 2.7 fold increase in average RCF lifetime with reduced scatter when a hard coating was deposited onto them. In this bearing configuration, the

Table 2: Initial TiN coated ß-Si_3N_4 Rolling Contact Screening Tests; TiN coating 0.3 μm thick*				
Sample Number	Drive Ball	Hertz Pressure, GPa (ksi)	RCF Lifetime, hrs	Comments
SNM50U03	uncoated ß-Si_3N_4	5.84 (851)	6.00	Drive ball fatigued
SNM50U07	uncoated ß-Si_3N_4	5.84 (851)	15.30	Two driven balls fatigued
TNSNU01	coated ß-Si_3N_4	5.84 (851)	30.40	One driven ball fatigued
TNSNU02	coated ß-Si_3N_4	5.84 (851)	23.65	One driven ball fatigued

* Driven Balls: Uncoated M50
Rotating speed of the top ball: 4800 rpm

baseline RCF lifetime value was 10.6 hours with a standard deviation, σ_{n-1}, of 6.6 hours, compared to an average lifetime of 27.0 hours and σ_{n-1} equal to 4.8 hours when coated. Therefore, not only did the coating enhance the RCF lifetime, it also improved its reliability.

As mentioned previously, the drive ball is more likely to fail in these rolling four-ball tests. In Table 2, the drive ball failed in only one of the four instances. This indicated that, even though the drive ball was undergoing more stress contact cycles, the high compressive stress in the coating prevented the propagation of cracks, and thus prevented the failure in the drive ball prior to that in the driven balls.

In order to examine this belief, an uncoated ß-Si_3N_4 was run against three M50 balls coated with 0.75 μm of TiN. When this system was tested using the same contact pressure and rotating speed as used for Table 2, **no fatigue was observed in over 95 hours of operation.** This result clearly indicated that the coating protected the driven balls, whereas this did not occur for the driven balls in Table 2. Similarly, a 0.75-μm-thick TiN coated M50 ball running against three uncoated ß-Si_3N_4 balls showed no fatigue in over 100 hours of operation using the same RCF test parameters. This is equivalent to 64.8 million contact stress cycles at 5.84 GPa (851 ksi) contact pressure without indication of failure.

4.3 Hybrid Bearing RCF Results-M50 Drive Ball

For the hybrid bearing system, it has been assumed that only a single component will be coated, and the more likely candidate will be the metal race. Thus, to more accurately simulate this system and produce fatigue in a reasonable time frame, the drive balls should be metal, and the coatings should be deposited on this substrate. RCF tests using uncoated M50 drive balls running against three uncoated ß-Si_3N_4 driven balls showed that the Hertz contact pressure and rotational speed used for Table 2 were inadequate to be used as an effective screening tool in the hybrid bearing RCF tests, so more severe conditions were examined. For a Hertz pressure of 6.90 GPa (990 ksi) and a rotational speed of 5400 rpm, a comparison of all three nitride coatings was possible, as shown in Table 3. Five sets of data are presented in the table: baseline, TiN coated, $Ti_{0.5}Al_{0.5}N$ coated at thicknesses of 0.5 and 1.0 μm, and CrN coated. On average, all of the coatings showed improvements over the baseline results, although the $Ti_{0.5}Al_{0.5}N$ coatings showed only marginal improvements except for the "TW" samples. In the tables, "T" and "B" indicate top and bottom cells, respectively, while "W" and "S" refer to two slightly different fixtures. The W fixture yields slightly more uniform coatings than the S fixture, which leads to longer RCF lifetimes. The five TiN coated samples are from two separate deposition cycles, denoted by "Ax" and "Bx" at the right of the sample number.

The baseline average RCF lifetime for the uncoated hybrid system in these tests was 8.30 hours with a standard deviation, σ_{n-1}, of 0.52 hours. When the driven balls are coated with TiN, the average RCF lifetime is at least 21.5 hours with σ_{n-1} equal to 16.07 hours. If the very low result of 1.8 hours is assumed to be due to an artifact of that ball's location within the basket and hence ignored, the average RCF lifetime for the TiN coatings is 26.5 hours with a σ_{n-1} of 13.5 hours. The 1.0-μm-thick $Ti_{0.5}Al_{0.5}N$ coatings demonstrated an average RCF lifetime of 11.1 hours and σ_{n-1} of 5.7 hours, while the 0.5-μm-thick $Ti_{0.5}Al_{0.5}N$ coatings had values of 10.7 and 2.2 for the average lifetime and standard deviation, respectively. The CrN coatings also performed well, with an average RCF lifetime of 16.5 hours and a 4.4 hour standard deviation. The average RCF lifetime values are also shown in Figure 11.

Table 3: RCF Testing of TiN, $Ti_{0.5}Al_{0.5}N$, and CrN Coated M50 Balls*			
Sample Number	Drive Ball, M50	RCF Lifetime, hours	Comments
M50A1	Uncoated	7.9	Fatigue on drive ball
M50A2	Uncoated	8.6	Fatigue on drive ball
TiNM50A1	TiN coated	38.0	No apparent fatigue pit; exceeded vibration level; 0.75 μm
TiNM50A2	TiN coated	1.8	0.75 μm
TiNM50B1	TiN coated	11.3	0.75 μm
TiNM50B2	TiN coated	37.7	0.75 μm
TiNM50B3	TiN coated	18.8	0.75 μm
TiAlN2A1	$Ti_{0.5}Al_{0.5}N$ coated	9.0	1.0 μm; TS
TiAlN2A2	$Ti_{0.5}Al_{0.5}N$ coated	6.9	1.0 μm; BS
TiAlN2A3	$Ti_{0.5}Al_{0.5}N$ coated	17.6	1.0 μm; TW
TiAlN2B1	$Ti_{0.5}Al_{0.5}N$ coated	9.1	0.5 μm; TS
TiAlN2B2	$Ti_{0.5}Al_{0.5}N$ coated	9.7	0.5 μm; BS
TiAlN2B3	$Ti_{0.5}Al_{0.5}N$ coated	13.2	0.5 μm; TW
CrNM50A1	CrN coated	18.1	0.75 μm
CrNM50A2	CrN coated	11.6	0.75 μm
CrNM50A3	CrN coated	19.9	0.75 μm

* Driven Balls: uncoated ß-Si_3N_4
Rotating speed of top ball: 5400 rpm
Hertz contact pressure: 6.80 GPa (990 ksi)

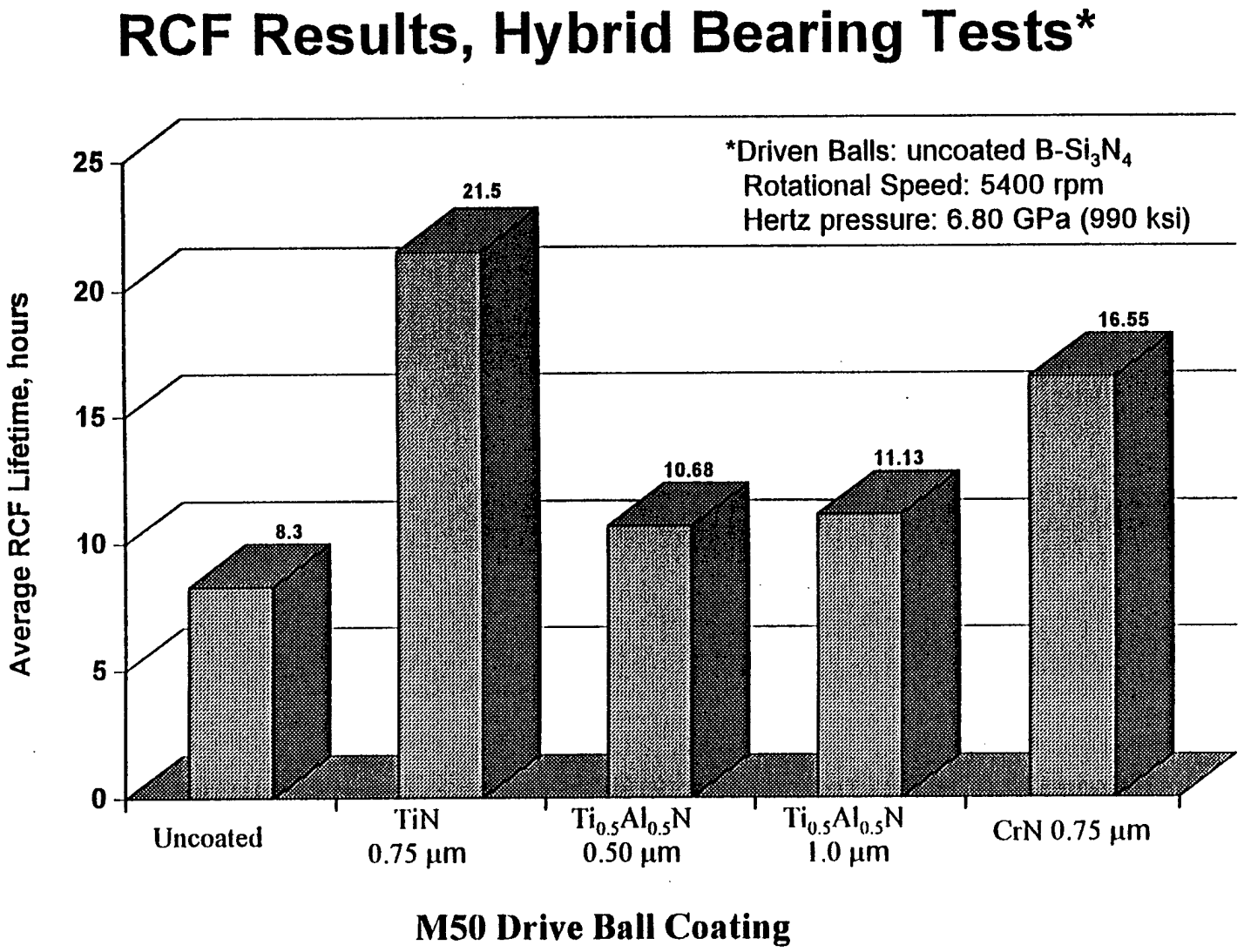

Figure 11. Summary of average rolling contact fatigue life results.

Improvement of RCF lifetimes by a factor of at least 2.6 was seen when the balls are coated with TiN, although there is a wide variation in results. As was shown with the $Ti_{0.5}Al_{0.5}N$ coatings, the location of the ball in the coating fixture can result in a less uniform coating. Since fatigue pits were observed on all of the coated drive balls, it may be that the M50 steel is failing due to the severity of the operating conditions. The Hertz contact pressure, for example, is several times larger than the yield strength of the M50. Closer examination of the coatings, the coating fixture, and the substrate material is required for a better understanding of these results.

5. TECHNOLOGY TRANSFER/COMMERCIALIZATION

Robert Price of Draper Labs provided 4.76-mm- (3/16-inch-) diameter ß-Si_3N_4 balls for coating and evaluation of the coating uniformity. These were coated with TiN in our single cathode sputtering unit using the conditions indicated in the coating section and returned for testing.

Draper Labs examined the size and sphericity of the coated balls, as well as the coating integrity when utilized in typical instrument bearing applications. The balls were matched to within 0.127 μm (5 μ-inch) before coating. After coating, the variance was 0.508 μm (20 μ-inch), but all balls remained spherical. Since all of the balls were coated in a single run, this indicates that a large variation in deposition rate exists within the basket. It also indicates that, regardless of position within the basket, the coating itself was uniform on each of the balls. On both accounts this information is encouraging. We have already suggested that some of the scatter in our RCF results may be due to variations in coating thickness within the basket. The uniformity of the coating on each of the balls, in spite of the thickness, does reinforce the belief that the technique is fundamentally sound.

Under actual application at Draper, some of the coatings scratched, dented, and/or peeled. This produced TiN transfer on the matching surfaces. As previously indicated, the coating conditions have produced coatings with a hardness of at least 2400 kgf·mm-2 and a critical load of 5.0 kgf for a 5-μm-thick film on an M2 steel substrate outside of a basket. Better critical loads for TiN coatings on ß-Si_3N_4 flats were obtained when 20-minute etch times were used. However, because of the design of the rotating device in the single-cathode sputtering system, such long etch times are not possible. Therefore, the Draper results are understandable given the need for longer etch times for ß-Si_3N_4 substrates in order to produce well-adhering coatings. This is the first time that samples other than the 0.5-inch-diameter RCF balls have been coated, and similar improvements should be attained if the proper coating conditions are used.

In another direct application, it is expected that the Naval Surface Warfare Division, Carderock Division, will have BIRL coat races and bearings for them. They will test the coated bearings as part of their Silent Bearing Program.

6. SUMMARY

The results from this program have been outstanding. Three nitride coatings have been successfully deposited on M50 and ß-Si_3N_4 substrates. Under extremely severe accelerated testing, each coating material has extended the average rolling contact fatigue lifetime for a complete four-ball bearing system. The greatest improvement was observed with 0.75-μm-thick TiN coatings, which extended the lifetime of the bearing system by a factor of 2.6 times relative to the uncoated system. Selected TiN coatings showed increases in RCF lifetime of a factor of 4.6. The CrN coatings showed improvements of a factor of 2.0, while the $Ti_{0.5}Al_{0.5}N$ coatings increased the lifetime by 1.3 times. This work has clearly indicated that improvements in RCF lifetime are achievable by coating either element of a hybrid bearing, while hinting at the possible improvements made by coating both elements. BIRL has demonstrated the ability to coat both the metal and ceramic components of hybrid bearings, even under the constraints of a basket. It is believed that even better results can be obtained on real components, because the limitations of baskets will not be imposed upon us. In addition, a great deal of development work has been performed in terms of understanding the effects of fixturing and coating thickness variation on the RCF lifetime. A technique has been devised to measure the thickness and uniformity of the coating. It has been demonstrated that hard coatings extend the RCF lifetime of standard and hybrid bearing components. It now remains to continue the development of procedures begun during this program to achieve its final goal: commercialization of improved bearings.

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